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(71) Applicant *(for all designated States except US)*: SAES GETTERS S.P.A. [IT/IT]; Viale Italia, 77, I–20020 Lainate (IT).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: MARTELLI, Daniele [IT/IT]; Viale Jenner, 76, I–20159 Milano (IT). URSO, Giuseppe [IT/IT]; Via Adamello, 4, I–20038 Seregno (IT).

(74) Agents: ADORNO, Silvano; Societa' Italiana Brevetti S.p.A., Via Carducci, 8, I–20123 Milano (IT) et al.

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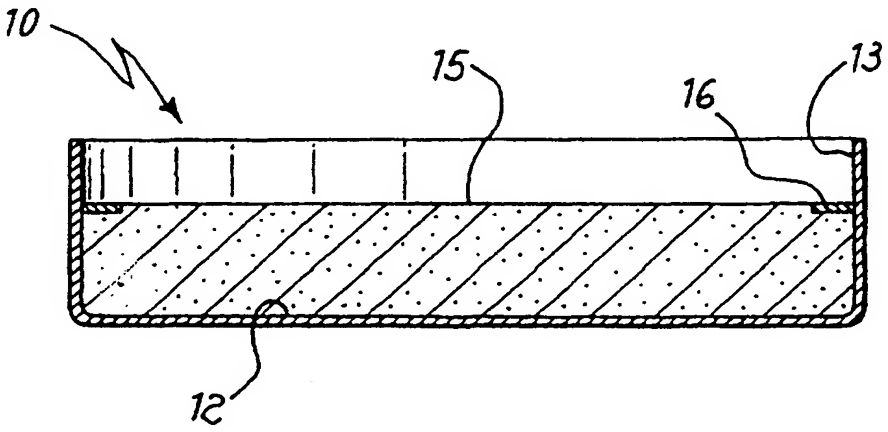

(57) Abstract

Evaporable getter devices (10) are described, with reduced loss of particles, formed of a metallic holder open at the top, containing a packet of powders of the mixture nickel–BaAl4, also comprising one or more crown–shaped metal members (16) placed in contact with the side walls (13) of the holder at the interface between these latter and the free surface (15) of the powders, so as to emerge with respect to such a free surface. A process for preparing these devices is also described.

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"EVAPORABLE GETTER DEVICE WITH REDUCED LOSS OF PARTICLES AND PROCESS FOR PRODUCING THE SAME"

The present invention deals with an evaporable getter device with reduced loss of particles and a process for producing the same.

The evaporable getter devices are known and since long used for the maintenance of the vacuum within TV picture tubes or computer displays.

As it is known, an evaporable getter device comprises a mixture of powders of nickel (Ni) and of the compound $BaAl_4$. The device, introduced in the picture tube before evacuation and sealing thereof, is then heated from the outside by means of radiofrequencies; during this operation, barium evaporates and deposits onto the inner walls of the picture tube in the form of a very thin film being the active element of the maintenance of vacuum.

Evaporable getter devices are described in a number of patents, among which for example the US patents 2,824,640; 2,907,451; 3,225,911; 3,381,805; 3,558,962; 3,719,433; 4,077,899; 4,134,041; 4,154,162; 4,665,343; 4,642,516; 4,961,040 and 5,118,988.

The devices disclosed by these patents are different from each other by construction of details, but are all essentially formed of a circular metallic holder (generally of steel) open at the upper side, wherein a seat of the mixture of powders is provided. In a plan top view of the device, such a seat may occupy all the surface available, thus resulting in a cylindrical volume to be filled with the powders, or may define a circular crown, generally of rectangular cross-section, whereby the volume available for the filling with the powders has the shape of an annular channel. In manufacturing these devices the desired quantity of powder mixture Ni-$BaAl_4$ is poured into the corresponding seat of the metallic holder and is compressed in this seat by means of a special shaped punch at pressure values of some tons per square centimeter, thus forming a compact packet. The punch has a diameter slightly smaller than the inner diameter of the holder, otherwise its lateral surface would seize up against the inner walls of the holder, with

consequent difficulties in removing the finished device from the punch; therefore there is a "clearance", generally of about 100 μm, between the side surface of the punch and the corresponding surfaces of the seat of the powders. The nickel powders used in the production of evaporable getter devices have generally a particle size of less than 60 μm, and those of $BaAl_4$ generally lower than 250 μm; consequently it may happen that grains of powder remain sticking to the side walls of the device, without being dragged downwards by the punch, and in any case the most external portion of the packet surface, corresponding to the "clearance" between punch and holder, proves to be very little compressed and of poor mechanical stability; these loose grains of powder or an inadequately compressed portion of the packet can give rise to a loss of particles from these devices at the inside of the picture tubes.

Particles of powder at the inside of a picture tube cause a number of problems: the particles may deposit onto the electron gun of the picture tube, thus altering its correct operation, or they may interfere with the high electric fields generated when the picture tube is working, or finally may obstruct some of the holes in the mask at the inside of picture tubes in the proximity of the screen, thus producing dark spots in the image formed thereon. Particularly felt by the manufacturers of picture tubes is the latter problem, essentially due to the particles having sizes of more than 50 μm.

The problem of the loss of particles is faced by the Italian patent application MI98A 001742 in the name of the Applicant. This document discloses evaporable getter devices wherein the free surface of the packet of powders is stabilized mechanically by the formation of a thin vitreous layer the composition of which comprises at least an oxide chosen among silicon oxide, germanium oxide, aluminum oxide and boron oxide. This method is efficient for solving the problem but burdensome from the production point of view as it increases the number of row materials and of operational steps required for manufacturing the device.

It is an object of the present invention to provide an evaporable getter device with reduced loss of particles, in particular those having a size greater than 50 μm.

Another object of the invention is that of providing a process for producing

an evaporable getter device with reduced loss of particles which does not require to make use of row materials or operation steps different from those typically used in this field of art.

These objects are achieved according to the present invention with an evaporable getter device comprising:

- an upperly open metallic container formed of an essentially flat bottom wall and at least a cylindrical side wall essentially perpendicular to said bottom wall;
- a packet of compressed powders of a mixture of nickel and $BaAl_4$ in the container, having a free upper surface;
- at least a metallic member the shape of which, in a top plan view, is a circular crown which is solid or has recesses and is such that at least one diameter of said metallic member is long as the diameter of at least one of said cylindrical side walls of the container, said metallic member being placed in contact with at least one of the cylindrical side walls of the container at the interface between the free surface of the powders and said side wall in such a way to emerge with respect to the free surface of the packet of powders and to be at least partially parallel to said surface.

The invention will be described in the following in some examples of possible embodiments with reference to the annexed drawings in which:

FIGURE 1 shows a first device according to the invention;

FIGURE 2 shows a cross-section view along a diameter of the device illustrated in Fig. 1;

FIGURE 3 shows a perspective view of a possible metallic member to be employed in the devices of the invention;

FIGURE 4 shows a perspective view of a second possible metallic member to be employed in the devices of the invention;

FIGURE 5a shows a cross-section view along a diameter of a device according to the invention comprising the metallic member of Fig. 4 in an intermediate step of production;

FIGURE 5b shows a finished device of the invention in a view similar to

that of Fig. 5a;

FIGURE 6 shows a second possible device of the invention;

FIGURE 7 shows a cross-section view along a diameter of a device of the type shown in Fig. 6;

FIGURE 8 shows a perspective view of a metallic member to be used in a device of the type shown in Fig. 6; and

FIGURE 9 schematically shows a system for measuring the loss of particles from evaporable getter devices.

With reference to Figs. 1 and 2 a first possible evaporable getter device 10 of the invention is shown, respectively in a perspective and a cross-sectional view, being of the type wherein the packet of powders of nickel and $BaAl_4$ occupies the whole surface of the device in a top plan view. This device comprises a container 11, generally formed by cutting and cold pressing a metal sheet; sheet steel AISI 304 is generally employed, with a thickness between about 0.08 and 0.35 mm. The container is formed so as to have an essentially flat bottom wall 12 and a cylindrical side wall 13 perpendicular to the wall 12, thus defining a seat of essentially cylindrical geometry for the packet 14 of mixed powders of nickel and $BaAl_4$. At the interface between the free surface 15 of packet 14 and the side wall 13 there is a metallic member, generally defined as member 16 in all the possible geometrical variations described in the following. Two possible embodiments of the member 16 are shown in Figs. 3 and 4. In any case this member, in a top plan view, has the shape of a circular crown, whereas its cross-section may be essentially flat as shown in Fig. 3, but preferably has the shape of Fig. 4, essentially defined by two main surfaces, one flat and one frustoconical, with the portion having a flat surface joined to the one with frustoconical surface along the smaller circumference of the latter. In both cases the longer diameter of the member 16 has the same length as the inner diameter of container 11, while its smaller diameter is slightly shorter than the longer one, generally 1 mm less, whereby the surface 15 results to be almost completely free and the barium evaporation is not obstructed.

Figs. 5a and 5b show a device of the invention in two different production

steps, as an example of use of member 16 of Fig. 4 having the preferred shape. As represented in Fig. 5a, member 16 is inserted into the container 11 after having introduced in the latter the desired quantity of powdered nickel and $BaAl_4$, before compressing the powders to form packet 14. During the compression step, the member 16, pushed downwards by a punch (not shown in the drawing) scrapes the inner surface of wall 13, dragging together with it the particles, if any, which on the contrary would not be contacted by the punch, the diameter of which, as stated above, is slightly shorter than the inner diameter of container 11. Furthermore member 16 is compressed into the packet 14 with the portion with planar surface emerging with respect to the free surface 15 of packet 14 and parallel thereto, with the outer edge contacting the side wall 13; with this arrangement member 16 occupies the area corresponding to the "clearance" between the side surface of punch and wall 13, thus eliminating the zone of powders poorly compressed existing in the known devices, which is the main source of loose particles. The metallic member 16 of Fig. 3, of flat geometry, is capable of carrying out the functions of cleaning the inner surface of the container and holding down the powders, but it could be detached from the device in consequence of knocks (for example during transportation); the preferred member 16, shown in Fig. 4, avoids these possible problems since during the compression undergoes a lateral deformation so that its frustoconical surface comes into contact with the inner portion of wall 13, thus resulting firmly retained in its position. The member 16 is made of metal: among the preferred metals there are steel, e.g. the same AISI 304 steel employed for the production of the container, or nickeled iron, nickel or its alloys.

Figs. 6 and 7 show, respectively in perspective and cross-sectional view, a second possible evaporable getter device 60 of the invention; in this type of device, in a plan top view, the packet of powders of nickel and $BaAl_4$ occupies only an outer zone, having a circular crown shape. In this case the device comprises a container 61, made of materials and with size similar to those adopted for the container 11; the seat for the powders is defined in this container by an essentially flat bottom wall 62 and two cylindrical walls 63 and 64, essentially

WHAT IS CLAIMED IS:

1. A method of fabricating an MIS transistor comprising the steps of:

a) providing a monocrystalline silicon substrate having a major surface,

b) cleaning said major surface,

c) placing the substrate in an atmosphere of a nitrogen compound at a pressure above one atmosphere,

d) heating the substrate and forming a nitride layer on said major surface,

e) placing the substrate and nitride layer in an atmosphere including oxygen (N_2O or O_2) at a pressure above one atmosphere,

f) heating the substrate and forming a silicon dioxide layer on said major surface under the nitride layer,

g) forming a gate electrode on the nitride layer, and

h) forming a source region and a drain region in the substrate abutting the major surface and aligned with the gate electrode.

2. The method as defined by claim 1 wherein step c) includes a nitrogen compound selected from the group consisting of nitric oxide (NO) and ammonia (NH_3).

3. The method as defined by claim 1 wherein step d) includes heating the substrate at a temperature in the range of 600° - 900°C for a period of time in the range of 5 - 20 minutes.

4. The method as defined by claim 1 wherein step f) includes heating the substrate at a temperature in the range of 600° - 900°C for a period of time in the range of 10 - 60 minutes.

5. The method as defined by claim 1 wherein step g) includes forming a layer of doped polysilicon on the nitride layer and etching the layer of polysilicon , nitride layer, and the silicon dioxide layer to form the gate electrode.

non-limiting examples show some embodiments designed to teach those skilled in the art how to put in practice the invention and to represent the best considered way for the accomplishment of the invention.

EXAMPLE 1

Ten devices of the type shown in Fig. 1 are manufactured. Each device comprises a container made of AISI 304 steel 0.18 mm thick and shaped so as to have an inner diameter of 20 mm and the side wall 13 of 4 mm in height. Into each device a mixture is introduced comprised of 550 mg of nickel in powder having a particle size of less than 60 μm and 550 mg of powdered $BaAl_4$ with particle size of less than 250 μm. A metallic member 16 having a planar-frustoconical geometry of the type shown in Fig. 4 is placed into each container. The devices are prepared by causing the powders in the container to be compressed by a punch under a pressure of 9700 kg/cm^2.

EXAMPLE 2 (COMPARATIVE)

Ten evaporable getter devices are prepared, absolutely similar to the devices of Example 1, except for non comprising the metallic member 16 of planar-frustoconical geometry placed at the intersection between the free surface of the packet of powders and the inner wall of the container.

EXAMPLE 3

This example describes a test relating to the particles being lost from the devices of the invention prepared as in Example 1.

A testing apparatus, schematically represented in Fig. 9, has been made, comprising a glass vessel 90 open at the top and connected, through a lower port and a rubber sleeve (not shown), to a glass conduit 91, in turn connected to a rotary pump 92. Along the conduit 91 there is placed a laser counter 93 of particles, model SYSTEM 3116 of the German firm PAMAS GmbH; this counter of particles is capable of measuring the number of particles passing through its laser ray, separated by grains size fraction. The vessel 90 is also connected, through an arm 94, to a motor 95.

To carry out the test, the ten samples prepared as in Example 1 are put into the container 90, the upper opening of which is closed by means of a filter which

keeps all the particles having a size greater than 0.3 μm. The pump 92 is operated so as to establish a gas flow towards the latter from the vessel 90, and subsequently the motor 95 is operated which, through arm 94, causes the vibration of vessel 90, so as to produce a loss of particles from the samples under test. At the same time of the motor starting up the particles counter 93 is put into action for detecting number and size of the particles lost from the test samples. The test lasts 4 minutes, because during previous tests for the setting up of the method it has been found that after such a duration the loss of particles is negligible. The test results are reported in Table 1, wherein the number of particles is given as measured by the counter 93 in correspondence with the fraction of particles size.

EXAMPLE 4 (COMPARATIVE)

The test of Example 3 is repeated on samples prepared as in Example 2. The results of this test are listed in Table 1.

Table 1

Particle size fraction	Number of lost particles Example 3	Number of lost particles Example 4
20 ÷ 50 μm	1570	2600
50 ÷ 70 μm	64	168
70 ÷ 100 μm	25	63
100 ÷ 150 μm	3	13

As shown by the results of the tests, the devices of the invention have a loss of particles clearly lower than the samples of the prior art; in particular, the loss reduction is of more than 60% for the particles having a size greater than 50 μm (and more than 75% for the particles of a size greater than 100 μm), which are those causing the biggest problems in the production of picture tubes.

CLAIMS

1. An evaporable getter device (10; 60) comprising:

- an upperly open metallic container (11; 61) formed of an essentially flat bottom wall (12; 62) and at least a cylindrical side wall (13; 63; 64) essentially perpendicular to said bottom wall;
- a packet of compressed powders (14; 65) of a mixture of nickel and $BaAl_4$ in the container, having a free upper surface (15; 66);

characterized by comprising at least a metallic member (16; 67) the shape of which, in a top plan view, is a circular crown which is solid or has recesses, such that at least a diameter of said metallic member is long as the diameter of at least one of said cylindrical side walls (13; 63, 64) of the container, said metallic member being placed in contact with at least one of the cylindrical side walls of the container in correspondence with the intersection between the free surface (15; 66) of the powders and said side wall (13; 63, 64) so as to emerge from the free surface of the packet of powders while being at least partially parallel to said surface.

2. A device (10) according to claim 1, comprising a container (11) having one single cylindrical side wall (13) perpendicular to said bottom wall (12) and one single metallic element (16) at the intersection between the free surface (15) of said packet of powders (14) and said single side wall.

3. A device according to claim 2, wherein said metallic member (16) has an essentially flat cross-section.

4. A device according to claim 2, wherein said metallic member (16) has a shape essentially defined by two main surfaces, one being flat and one of frustoconical shape, with the portion of flat surface type being connected to that of frustoconical shape surface along the smaller circumference of the latter.

5. A device according to claim 2, wherein the greater diameter of said metallic member (16) has the same length as the inner diameter of the container (11).

6. A device (60) according to claim 1, comprising a container (61) having an outer cylindrical side wall (63) and an inner cylindrical side wall (64) both perpendicular to said bottom wall (62) and at least a metallic member (67) at the

intersection between the free surface (66) of said packet of powders (65) and at least the outer cylindrical side wall (63).

7. A device according to claim 6, wherein a single metallic member (67) is located in correspondence with the outer wall (63).

8. A device according to claim 7, wherein said single metallic member (67) has an essentially flat cross-section.

9. A device according to claim 7, wherein said single metallic member (67) has a shape essentially defined by two main surfaces, a flat one and a frustoconical one, with the flat surface portion connected to the frustoconical one along the smaller circumference of the latter.

10. A device according to claim 6, wherein a single metallic member (67) is provided in correspondence of both the outer wall (63) and the inner wall (64).

11. A device according to claim 10, wherein said metallic member (67) is integrally formed of a single piece obtained by cutting and possible shaping from a single metal sheet and comprises two circular crown-shaped portions being in contact with the cylindrical walls (63, 64) connected to each other by one or more radial portions (68).

12. A device according to claim 6, wherein two metallic members (67) are placed in correspondence with the outer wall (63) and the inner wall (64) respectively.

13. A device according to claim 12, wherein each one of the metallic members (67) having a circular cross-section, one independently from the other, can have a planar cross-section or a shape essentially defined by two main surfaces, a flat one and a frustoconical one.

14. A device according to claim 13, wherein the metal member (67), of frustoconical shape, is in contact with the outer wall (63), and the planar and frustoconical portions are mutually connected along the smaller circumference of the latter.

15. A device according to claim 13, wherein the metallic member (67), of planar-frustoconical shape, is in contact with the inner wall (64) and the planar portion and the frustoconical portion are mutually connected along the greater circumference of the latter.

16. A process for manufacturing the device of claim 1, comprising the following steps:

- providing a container (11; 61) of powders, having an essentially flat bottom surface (12; 62) and one (13) or two (63, 64) cylindrical side surfaces perpendicular to said bottom surface;
- introducing into the container a pre-defined quantity of a mixture of powders of nickel and compound $BaAl_4$;
- placing in the container, above the free surface of the powders (15; 66) and in contact with one or both the cylindrical side walls of said container, at least one metallic member (16; 67) the shape of which, in a plan view, is a circular crown being solid or having recesses, such that at least one diameter of said metallic member has the same length of diameter of at least one of the cylindrical side walls of said container; and
- compressing within said container the powders and said at least one metallic member (16; 67) by means of a shaped punch.

Fig.1

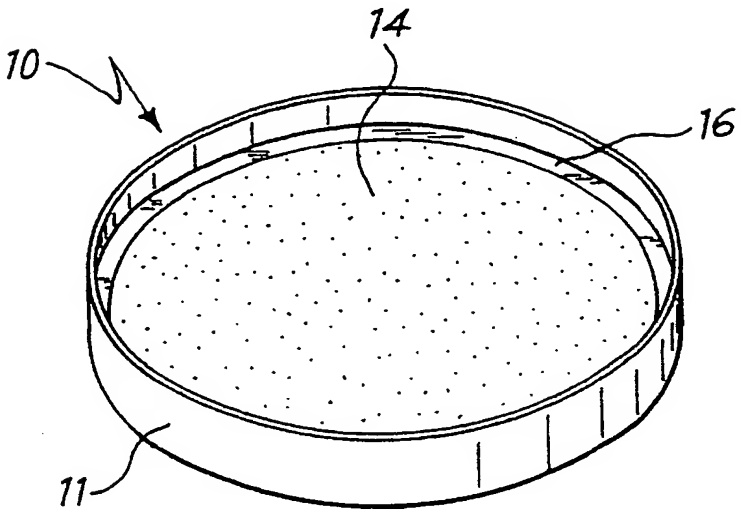
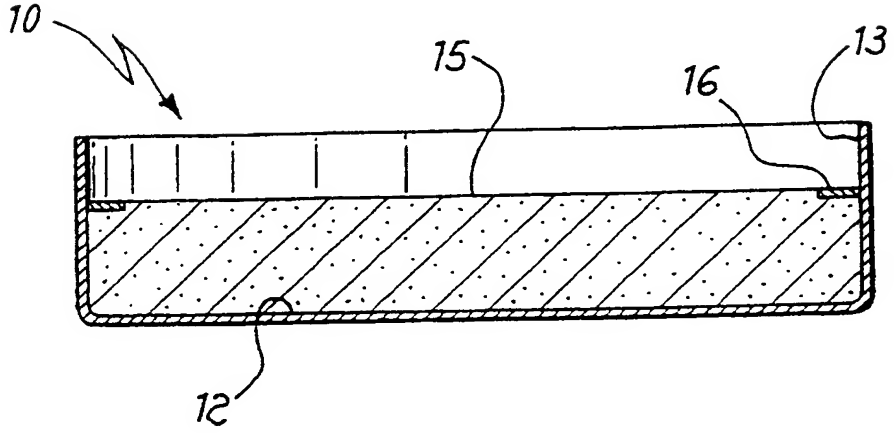


Fig.2



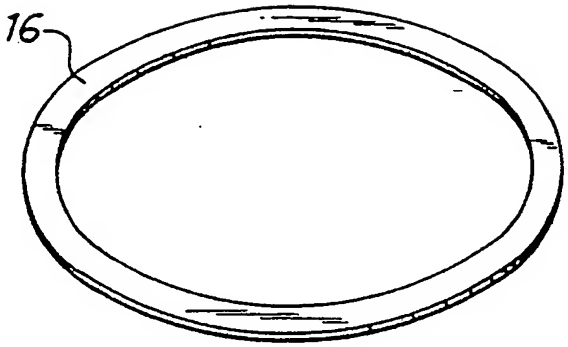


Fig.3

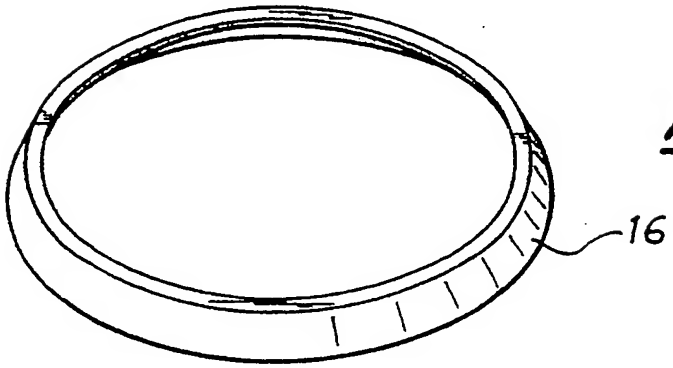


Fig.4

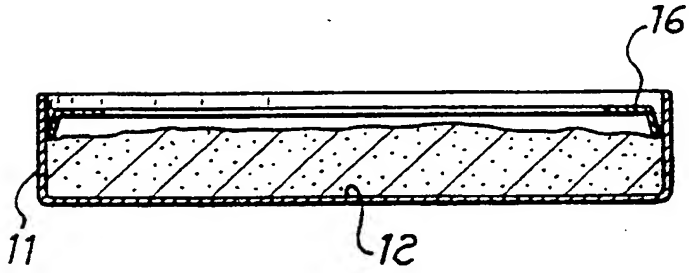


Fig.5a

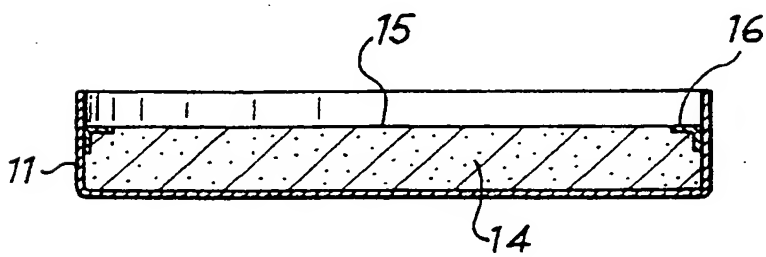


Fig.5b

Fig.6

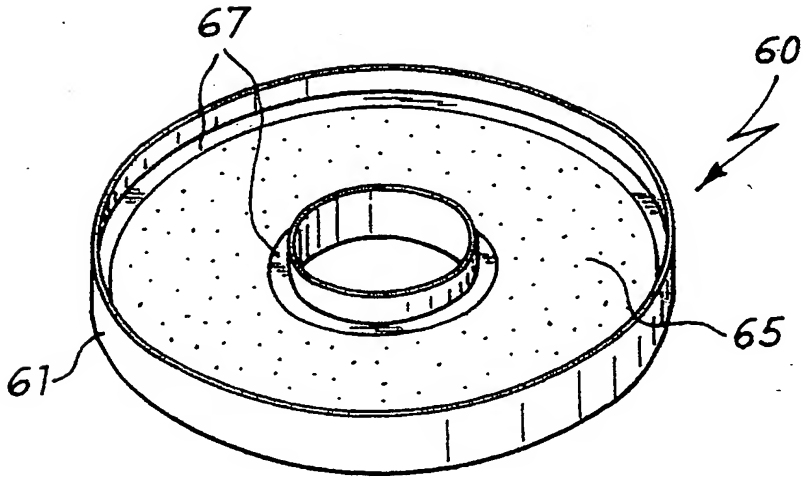


Fig.7

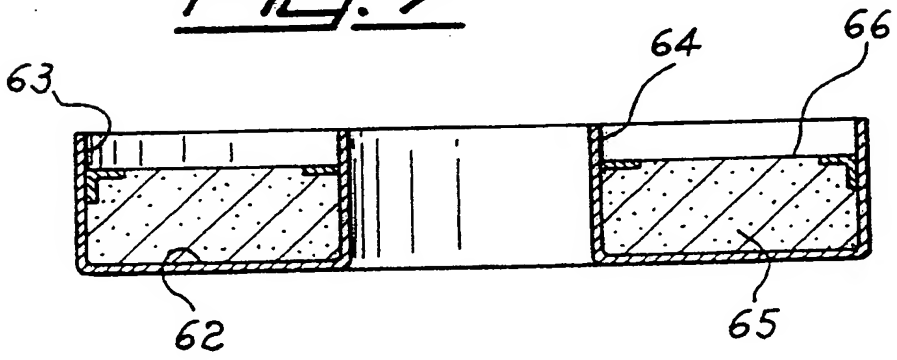
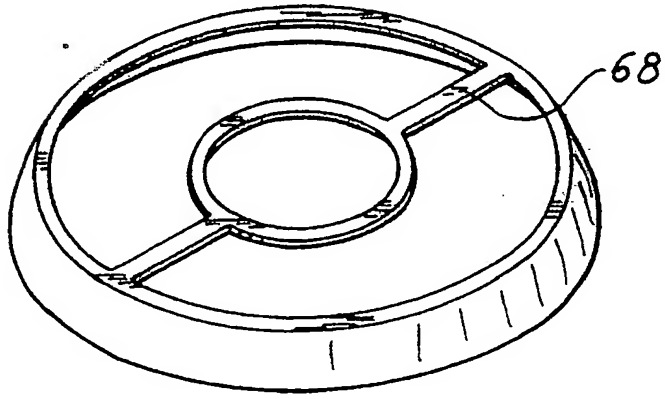


Fig.8



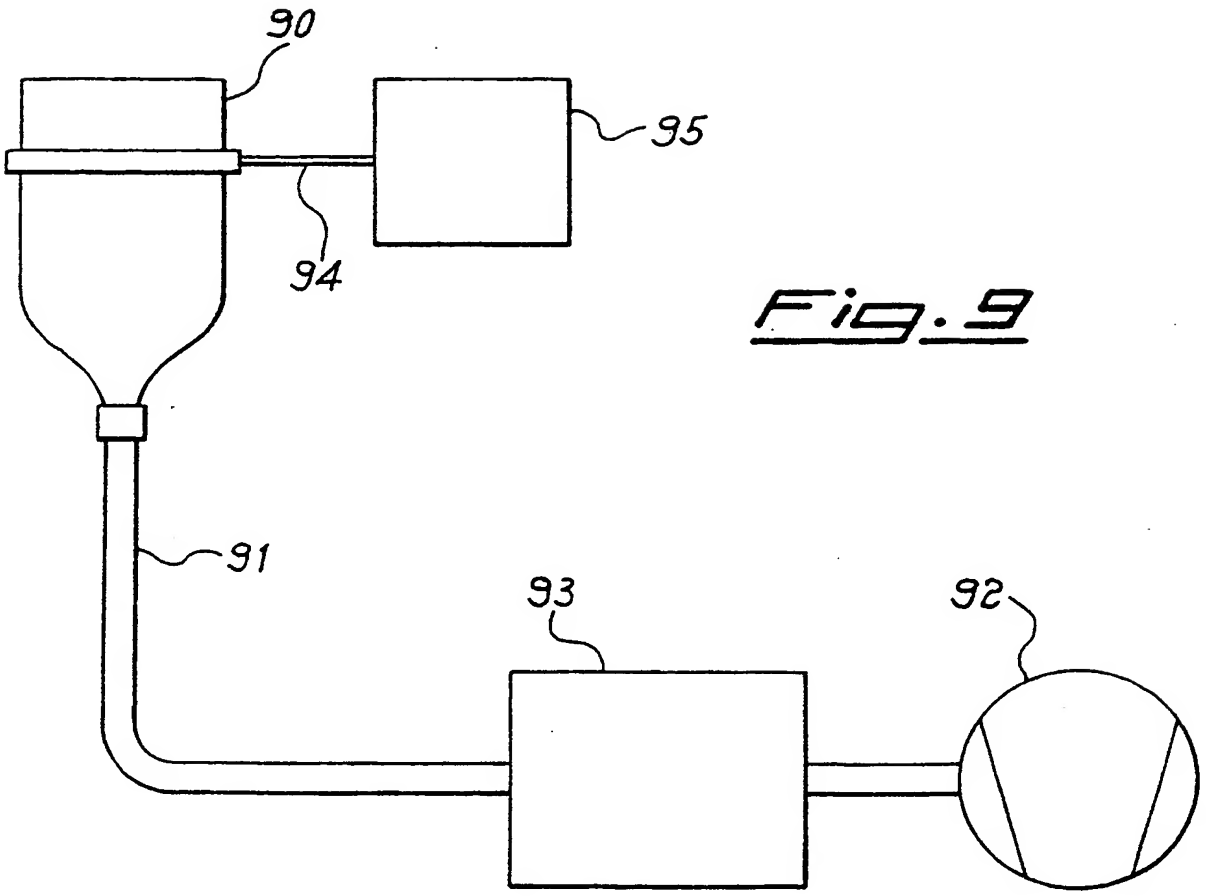


Fig. 9

INTERNATIONAL SEARCH REPORT

Int. .tional Application No
PCT/IT 99/00359

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 H01J7/18

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 H01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 853 328 A (GETTERS SPA) 15 July 1998 (1998-07-15) claims 1-15 ---	1
A	GB 736 475 A (SOCIETA APPARECHI ELETTRICI E SCIENTIFICI) claims 1-9; figure 7 ---	1,16
A	WO 97 50107 A (PHILIPS ELECTRONICS NV ;PHILIPS NORDEN AB (SE)) 31 December 1997 (1997-12-31) ---	
A	GB 1 186 581 A (S.A.E.S. GETTERS S.P.A.) 2 April 1970 (1970-04-02) ---	
A	US 3 560 788 A (REASH CLAIR ET AL) 2 February 1971 (1971-02-02) ---	

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Van den Bulcke, E

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INTERNATIONAL SEARCH REPORT

Int. ..ional Application No
PCT/IT 99/00359

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